



# **The Development of Environmental Barrier Coating Systems for SiC-SiC Ceramic Matrix Composites: Environment Effects on the Creep and Fatigue Resistance**

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in supporting the coating procesing



## Outline

- **Environmental barrier coating system development: needs, challenges and limitations**
- **Advanced environmental barrier coating systems (EBCs) for CMC airfoils and combustors**
  - NASA EBC systems and material system evolutions
  - Current turbine and combustor EBC coating emphases
  - Advanced development, processing, testing and modeling
  - EBC and EBC bond coats: recent advances
- **Design tool and life prediction of coated CMC components**
- **Advanced CMC-EBC rig demonstrations**
- **Summary and future directions**

# Durable Environmental Barrier Coating Systems for Ceramic Matrix Composites (CMCs):

Enabling Technology for Next Generation Low Emission, High Efficiency and Light-Weight Propulsion



## — NASA Environmental barrier coatings (EBCs) development objectives

- Help achieve future engine temperature and performance goals
- Ensure component system durability – working towards prime reliant coatings
- Establish database, design tools and coating lifing methodologies
- Improve technology readiness



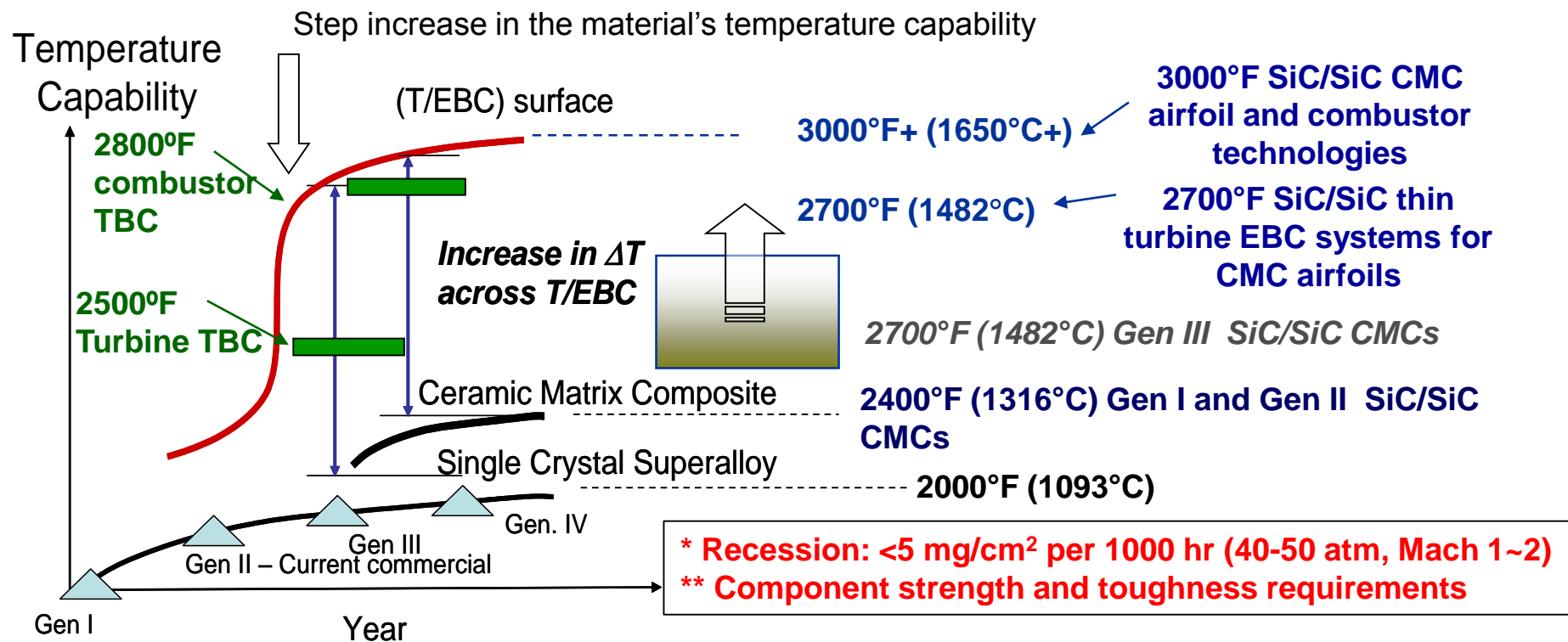
Fix Wing Subsonic Aircraft



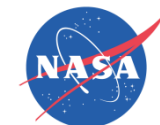
Supersonics Aircraft

# NASA Environmental Barrier Coating Development Goals

- Emphasize temperature capability, performance and durability
  - Low silica activity silicate and high stability/high toughness oxide system developments
- Develop innovative coating technologies and life prediction approaches
- 2700°F (1482°C) EBC bond coat technology for supporting next generation
- 2700-3000°F (1482-1650°C) turbine and CMC combustor coatings
  - Meet 1000 hr for subsonic aircraft and 9,000 hr for supersonics/high speed aircraft hot-time life requirements



# Evolution of NASA EBC Technology for SiC/SiC Ceramic Matrix Composites: Current State of the Art



## – Advanced EBC system developments

	Gen I (EPM) 1995-2000 R&D Award	Gen II (UEET) 2000-2004	Gen III (UEET) 2000-2005 R&D Award (2007)	Gen IV (FAP) 2005-2011 R&D Award (2007) coating turbine development	Gen V (FAP - ERA) 2007 – 2012 to present	Gen VI-VII (FAP) 2009 – present Patent 13/923,450 PCT/US13/46946
Engine Components:	Combustor	Combustor/ (Vane)	Combustor/ Vane	Vane/ Blade	- Vane/Blade EBCs - Equivalent APS combustor EBCs	Airfoil components
Top Coat:	BSAS (APS)	RE <sub>2</sub> Si <sub>2</sub> O <sub>7</sub> or RE <sub>2</sub> SiO <sub>5</sub> (APS)	- (Hf, Yb, Gd, Y) <sub>2</sub> O <sub>3</sub> - ZrO <sub>2</sub> /HfO <sub>2</sub> +RE silicates - ZrO <sub>2</sub> /HfO <sub>2</sub> +BSAS (APS and EBPVD)	RE-HfO <sub>2</sub> -Alumino silicate  (APS and/or 100% EB- PVD)	RE-HfO <sub>2</sub> -X advanced top coat RE-HfO <sub>2</sub> -graded Silica (EB-PVD)	Advanced EBC
Interlayer:	--	--	RE-HfO <sub>2</sub> /ZrO <sub>2</sub> - aluminosilicate layered systems	Nanocomposite graded oxide/silicate	Gen IV interlayer not required (optional)	
EBC:	Mullite+ BSAS	BSAS+Mullite	RE silicates or RE-Hf mullite	RE doped mullite-HfO <sub>2</sub> or RE silicates	Multi-component RE silicate systems	Multicomponent /self grown
Bond Coat:	Si	Si	Oxide+Si bond coat	HfO <sub>2</sub> -Si-X, doped mullite/Si SiC nanotube	Optimized Gen IV HfO <sub>2</sub> -Si-X bond coat 2700°F bond coats	RE-Si+X systems
Thickness	10-15 mil	10-15 mil	15-20 mil	10 mil	5 mil	1 -3 mils
Surface T:	Up to 2400°F	2400°F	3000°F/2400CMC	2700°F/2400F CMC	3000°F	
Bond Coat T:	Limited to 2462°F	Limit to 2462°F	Limit to 2642°F	Proven at 2600°F +;	2700°F (2011 goal)	
<b>Challenges overcome by advancements:</b>		Improved phase stability, recession resistance of top coat		Increased phase stability and toughness targeting 2700°F		Advanced compositions & processing for thinner coatings, higher stability and increased toughness

## Fundamental Recession Issues of CMCs and EBCs

### - Recession of Si-based Ceramics

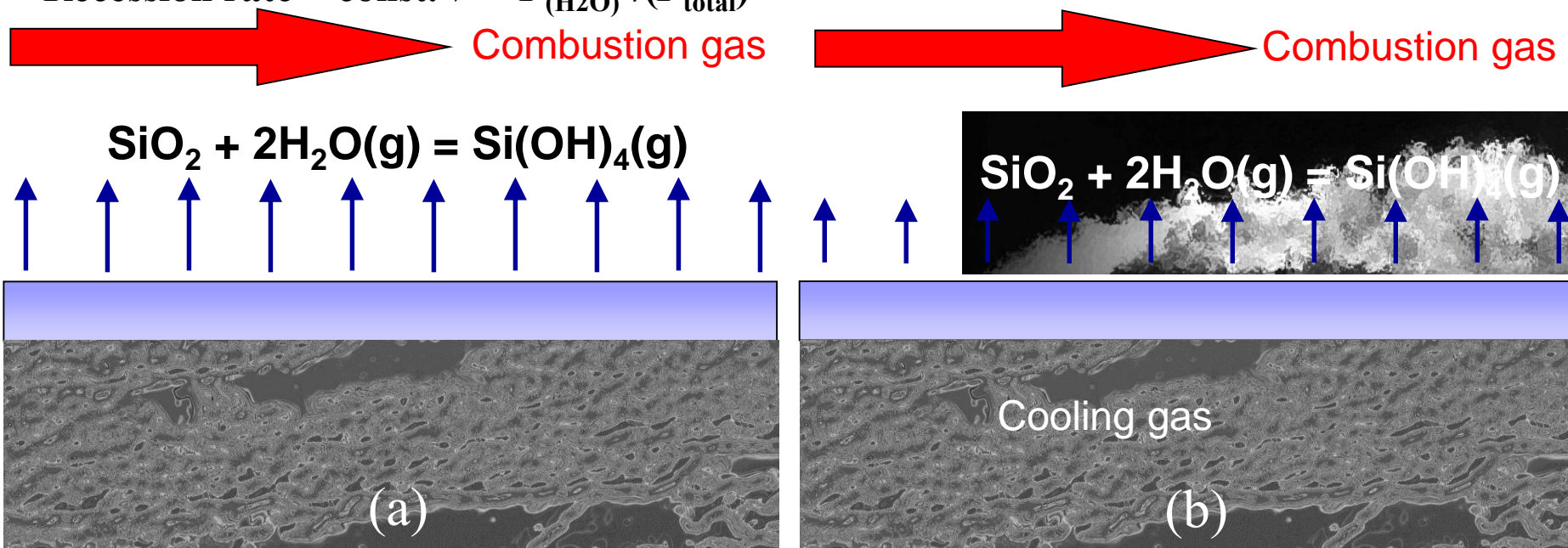
(a) Convective; (b) Convective with film-cooling

- Low  $\text{SiO}_2$  activity EBC system development emphasis

### - Advanced rig testing and modeling

More complex recession behavior of CMC and EBCs in High Pressure Burner Rig

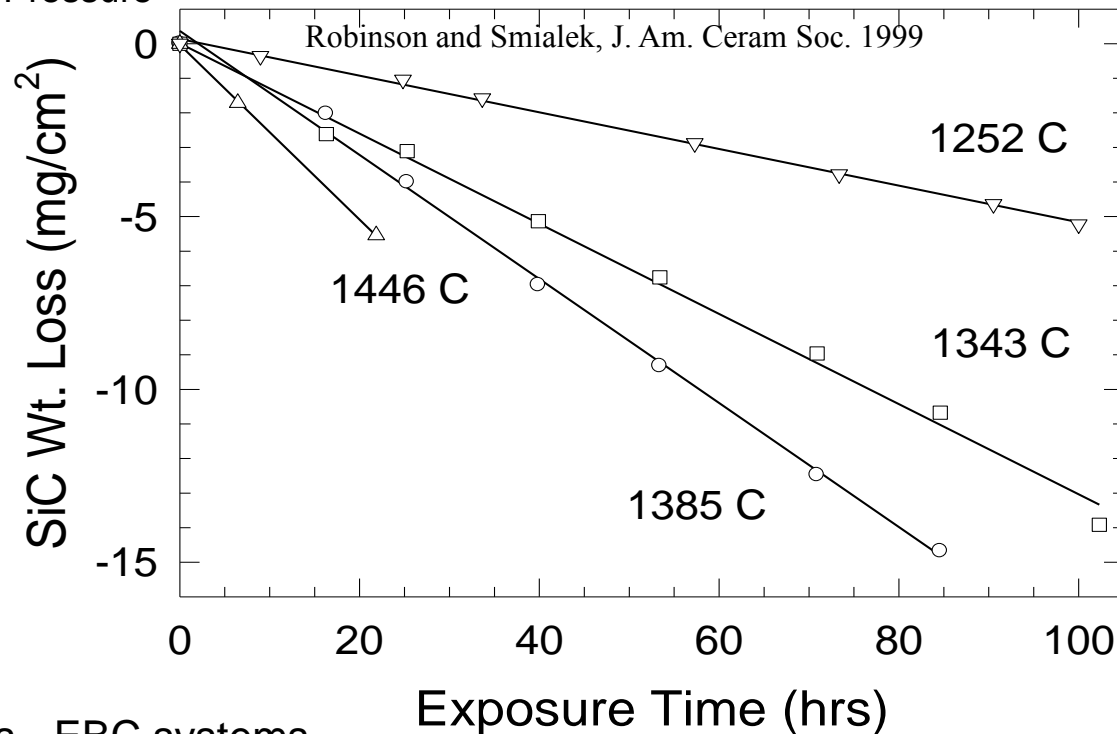
$$\text{Recession rate} = \text{const. } V^{1/2} P_{(\text{H}_2\text{O})}^2 / (P_{\text{total}})^{1/2}$$



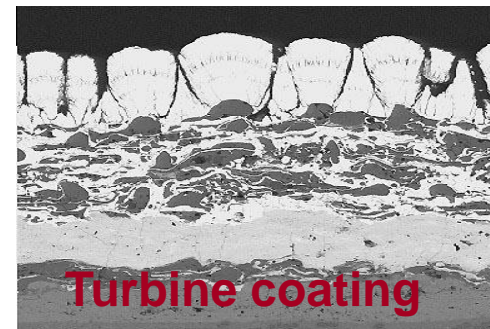
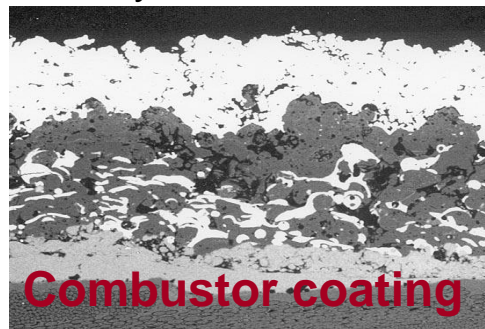
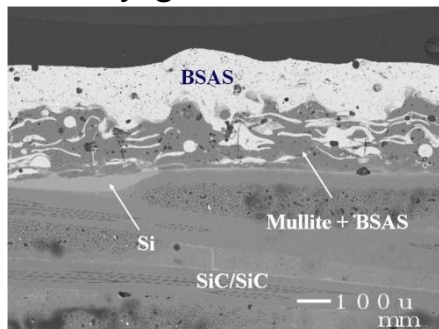


# Fundamental Recession Issues of CMCs and EBCs - Continued

Weight Loss of SiC in High Pressure  
Burner Rig  
6 atm 20 m/s



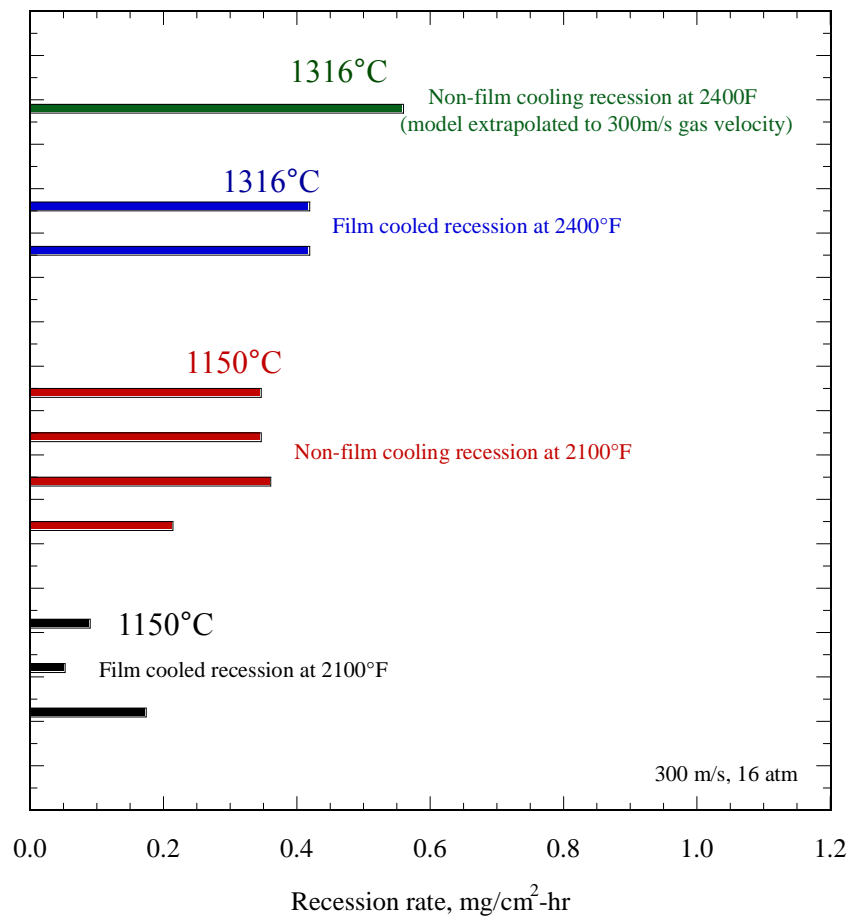
- Early generation coatings - EBC systems



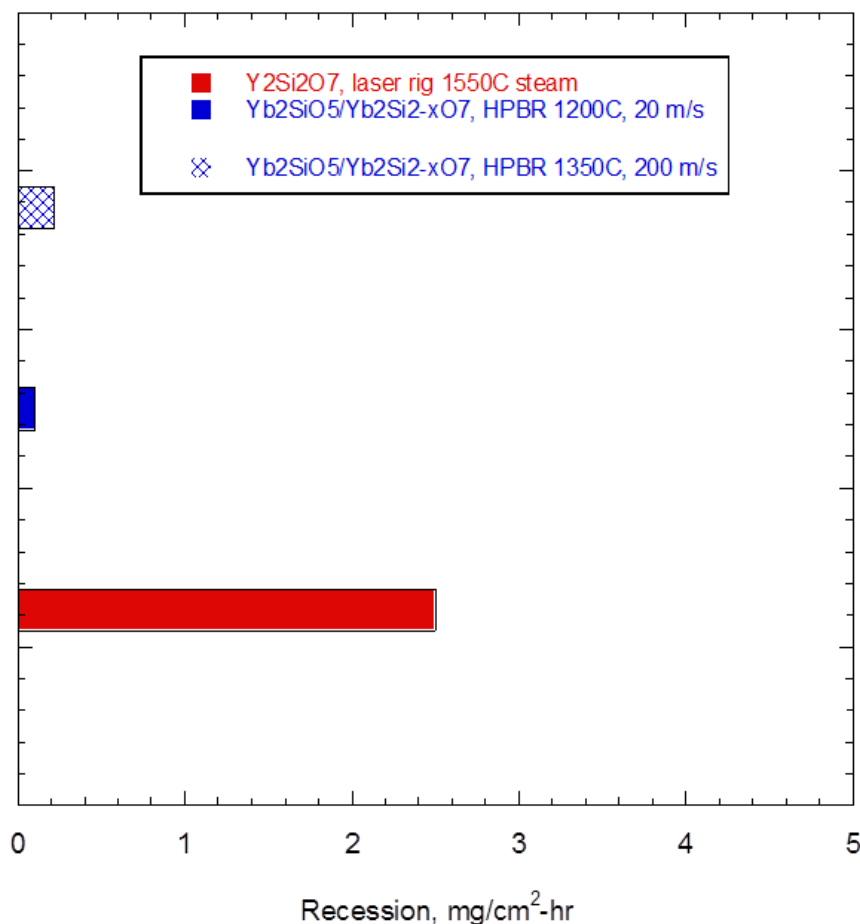


# SiC/SiC CMC and EBC Recession Kinetics Determined for CMCs-EBCs in High Pressure Bruner Rig and Laser Steam Rig Testing

— Determined recession under complex, and realistic simulated turbine conditions



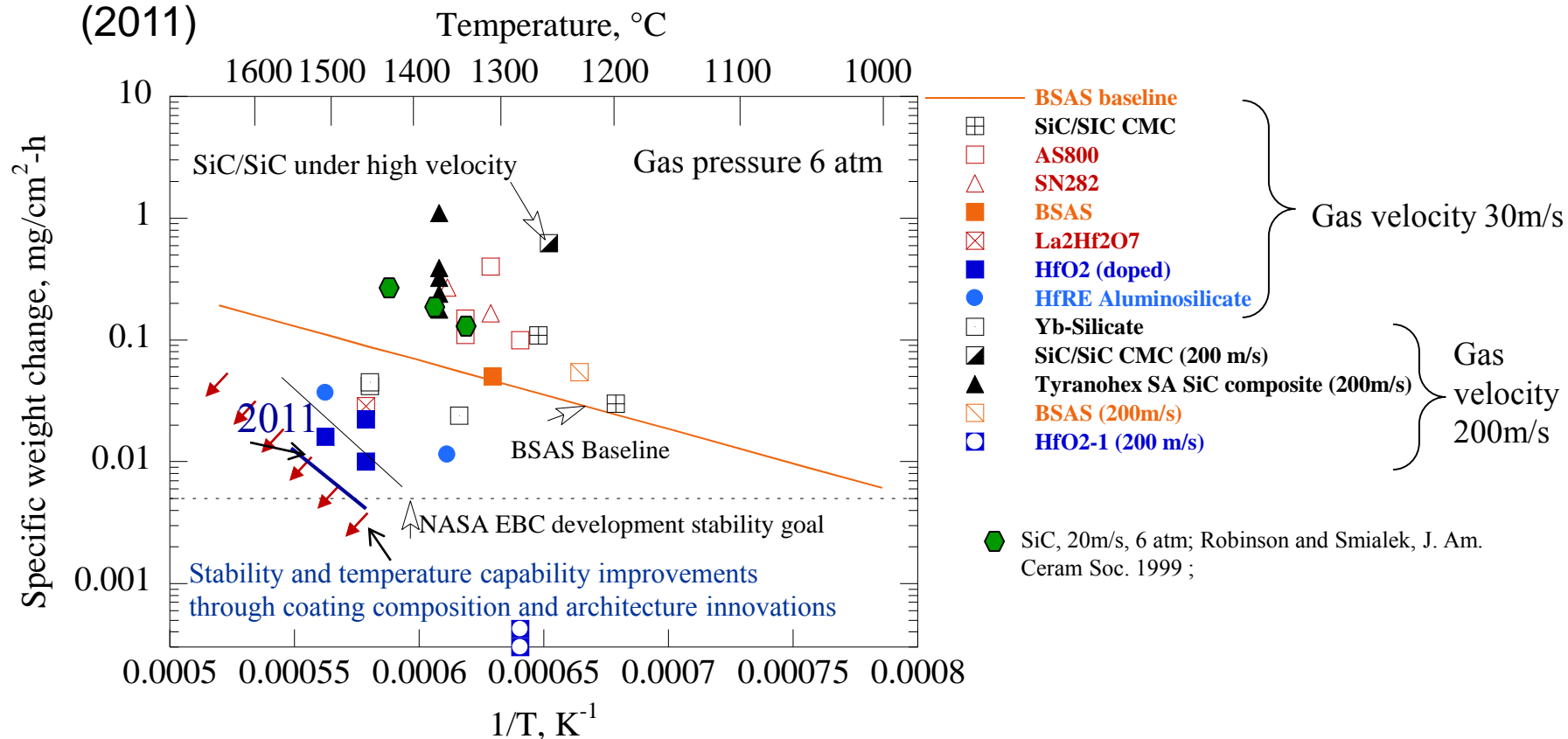
High temperature recession kinetics for film-cooled and non-film cooled Gen II SiC/SiC CMCs



Examples of environmental barrier coating recession in laboratory simulated turbine engine conditions

## Environmental Stability of Selected Environmental Barrier Coatings Demonstrated in NASA High Pressure Burner Rig

- EBC stability evaluated on SiC/SiC CMCs in high velocity, high pressure burner rig environment
- Advanced EBC recession met NASA Fundamental Aeronautics Project goals (2011)



Stability of selected coatings systems

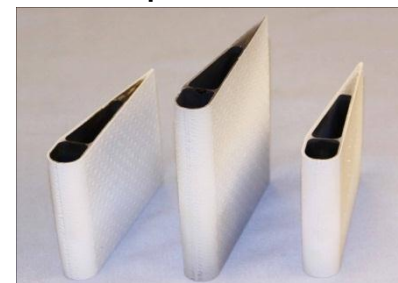
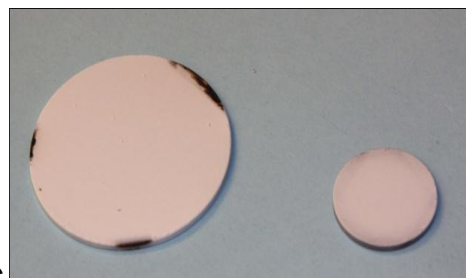
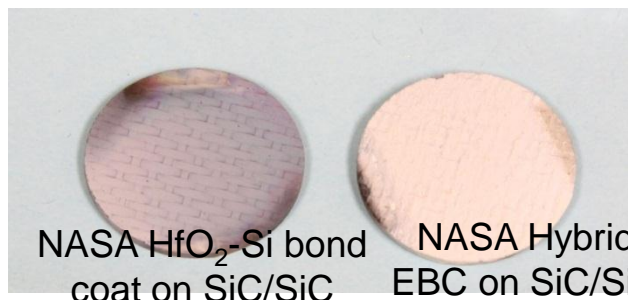


## NASA EBC Developments under NASA Programs

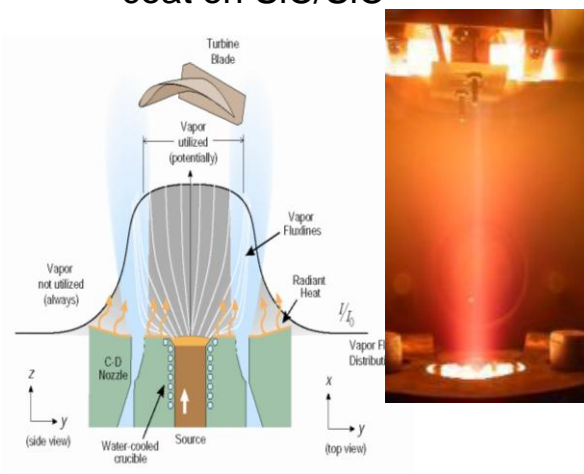
- Focus on improving technology readiness level (TRL), high stability multicomponent  $\text{HfO}_2$  or  $\text{ZrO}_2$ ,  $\text{HfO}_2\text{-RE}_2\text{O}_3\text{-SiO}_2/\text{RE}_2\text{Si}_{2-x}\text{O}_{7-2x}$  / environmental barrier/environmental barrier seal coat, with 2600°F+  $\text{HfO}_2\text{-Si}$  and 2700°F+ rare-earth silicon (silicides) bond coats
  - Calcium Magnesium Alumino-Silicate (CMAS) resistance addressed for the composition developments
- Developed and evaluated EB-PVD/plasma spray hybrid turbine airfoil coatings
  - Efforts in developing turbine EBC coatings with Directed Vapor Technologies
- Developed Triplex Pro and DVC based combustor EBC processing with Sulzer/Oerlikon Metco and Praxair
  - Efforts in developing new EBC coating composition powders with Oerlikon Metco
  - Efforts in developing EBC bond coat powders with Oerlikon Metco
  - Efforts in EBCs and DVM/DVC coatings in collaborations with Praxair
- Processing optimizations for improved plasma sprayed coating powders composition controls and coating processing
  - Plasma sprayed EBC coatings using Triplex Pro (with Mitch Dorfman)
  - Optimizing/developing commercial  $\text{HfO}_2\text{-Si}$  based series bond coats with Sulzer/Oerlikon (with Mitch Dorfman)
  - NASA in-house Plasma spray – Physical Vapor Depositions process optimization
- Developing 2000°F capable oxidation/fretting wear resistant coatings (Ti-Si-Cr/Ta-CN systems and NiAl/NiAl+Cr/high toughness oxide/silicate systems)

# Development and Processing of Directed Vapor Electron Beam - Physical Vapor Deposition (EB-PVD)

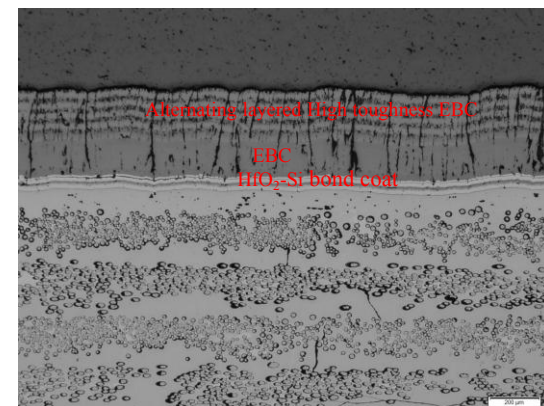
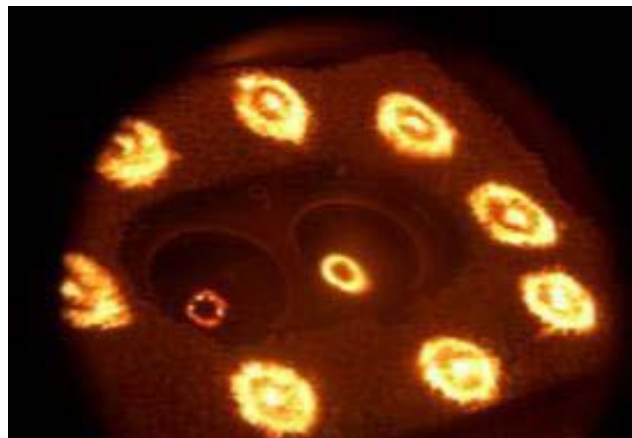
- NASA programs in supporting processing developments and improvements with Directed Vapor Technologies International, Inc.
  - Multicomponent thermal and environmental barrier coating vapor processing developments
  - High toughness erosion resistant turbine coatings
  - Affordable manufacture of environmental barrier coatings for turbine components



Advanced multi-component and multilayer turbine EBC systems



Directed Vapor Processing systems

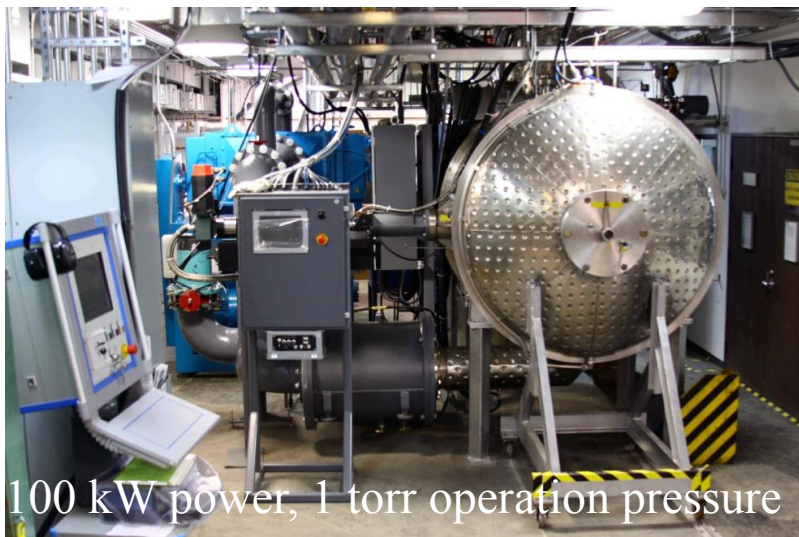
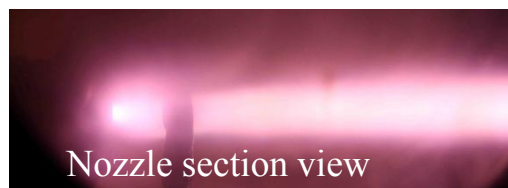


Processed EBC system

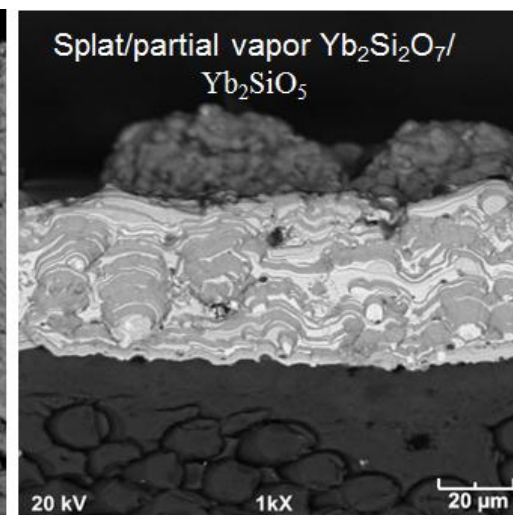
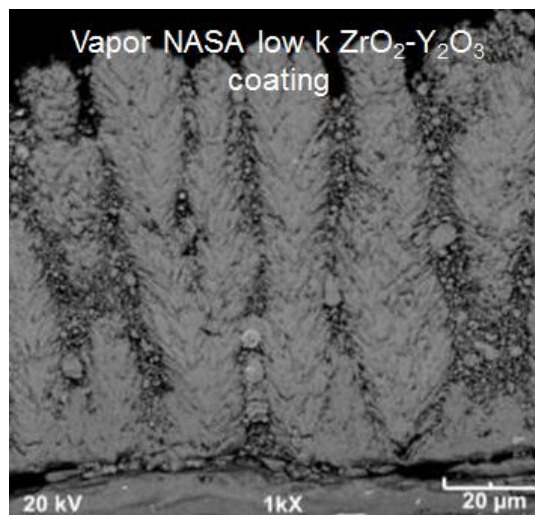


# Plasma Sprayed-Physical Vapor Deposition (PS-PVD) Processing of Environmental Barrier Coatings

- NASA PS-PVD and PS-TF coating processing using Sulzer newly developed technology
  - High flexibility coating processing – PVD - splat coating processing at low pressure (at ~1 torr)
  - High velocity vapor, non line-of-sight coating processing for complex-shape components
  - Emphasis on fundamental process and powder composition developments for EBC depositions



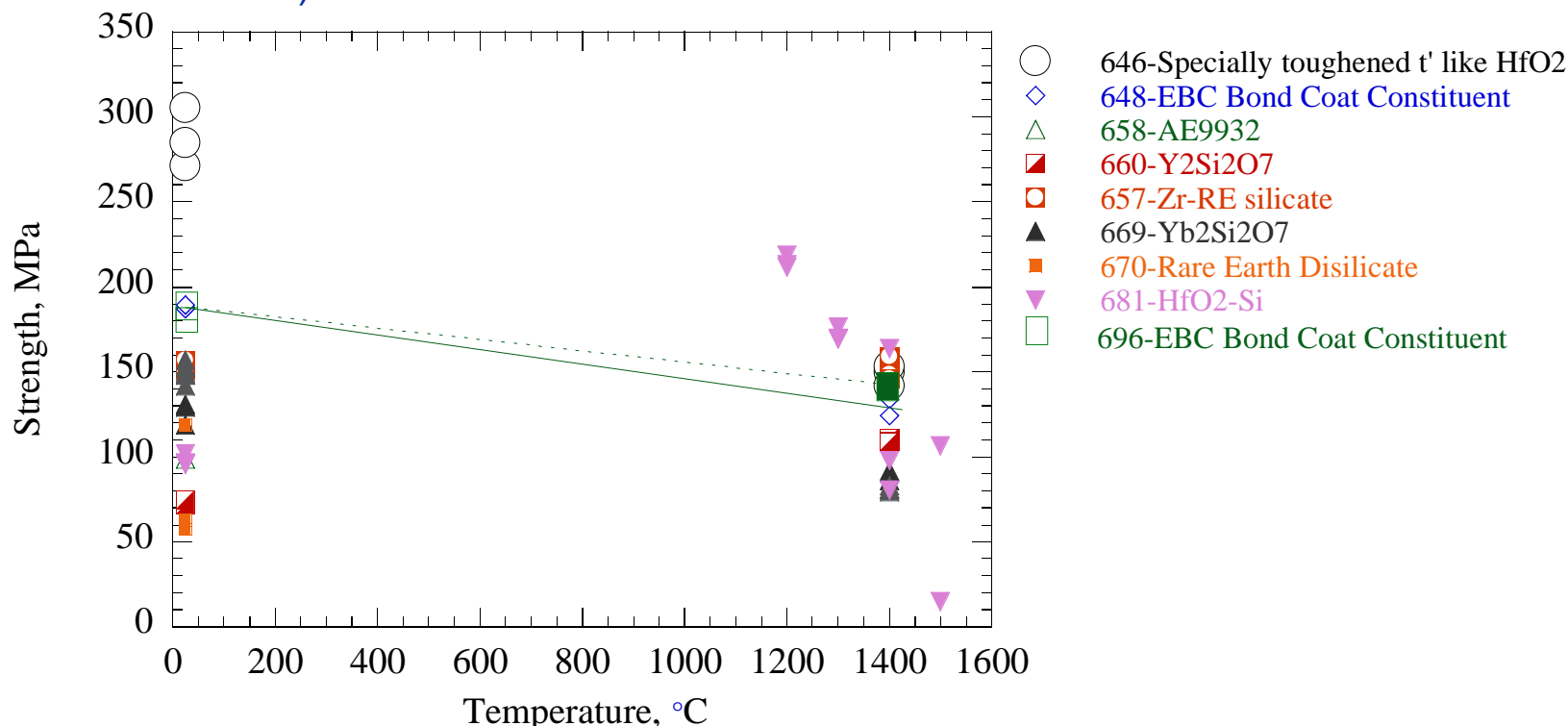
NASA hybrid PS-PVD coater system



Processed coater system

## Advanced EBC Coating Material Strength Evaluations

- EBC and bond coat constituents are designed with high strength to achieve the ultimate coating durability
  - Advanced EBC 150-200 MPa strength achieved at high temperature
  - Toughness 3-4 MPa m<sup>1/2</sup> achieved at room temperature
- HfO<sub>2</sub>-Si based systems showed promising strength and toughness
  - 100 – 250 MPa strength
  - Toughness 2-3 MPa m<sup>1/2</sup> achieved (Room Temperature, compared to silicon 0.8-0.9 MPa m<sup>1/2</sup>)

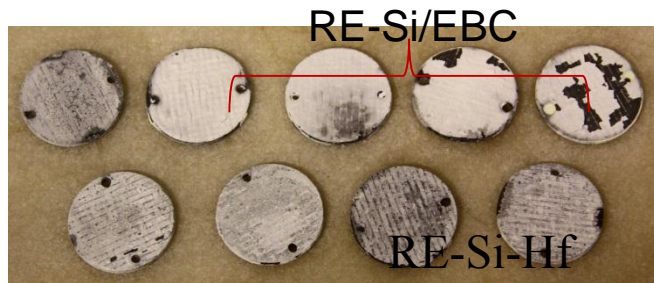


# Advanced EBC Bond Coats for Turbine Airfoil and Combustor EBCs Developed

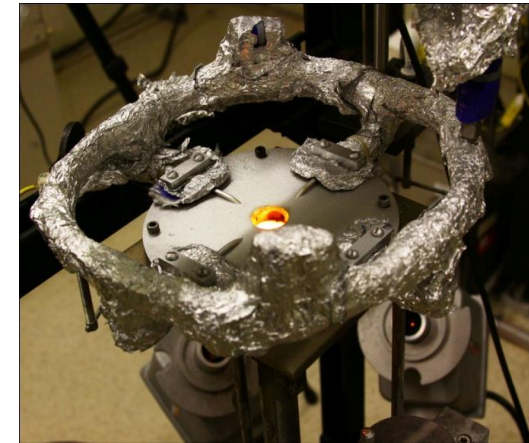
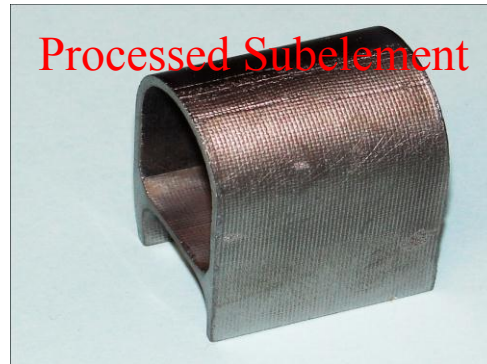


– Patent Application 13/923,450 PCT/US13/46946

- 1500°C (2700°F) capable RESiO+X(Ta, Al, Hf, Zr ...) EBC bond coat compositions and related composite coatings developed for combustor and turbine airfoil applications
- The bond coat systems demonstrated durability in the laser high heat flux rig in air and steam thermal gradient cyclic testing
- The bond coatings also tested in thermal gradient mechanical fatigue and creep rupture conditions



Processed Subelement



Steam heat flux test rig of the bond coat

Selected  
Composition Design  
of Experiment  
Furnace Cyclic Test  
Series 1500°C, in air,  
Demonstrated 500hr  
durability

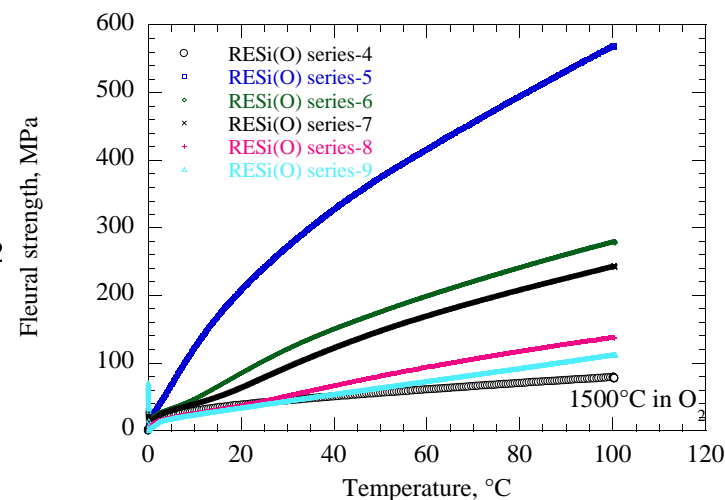
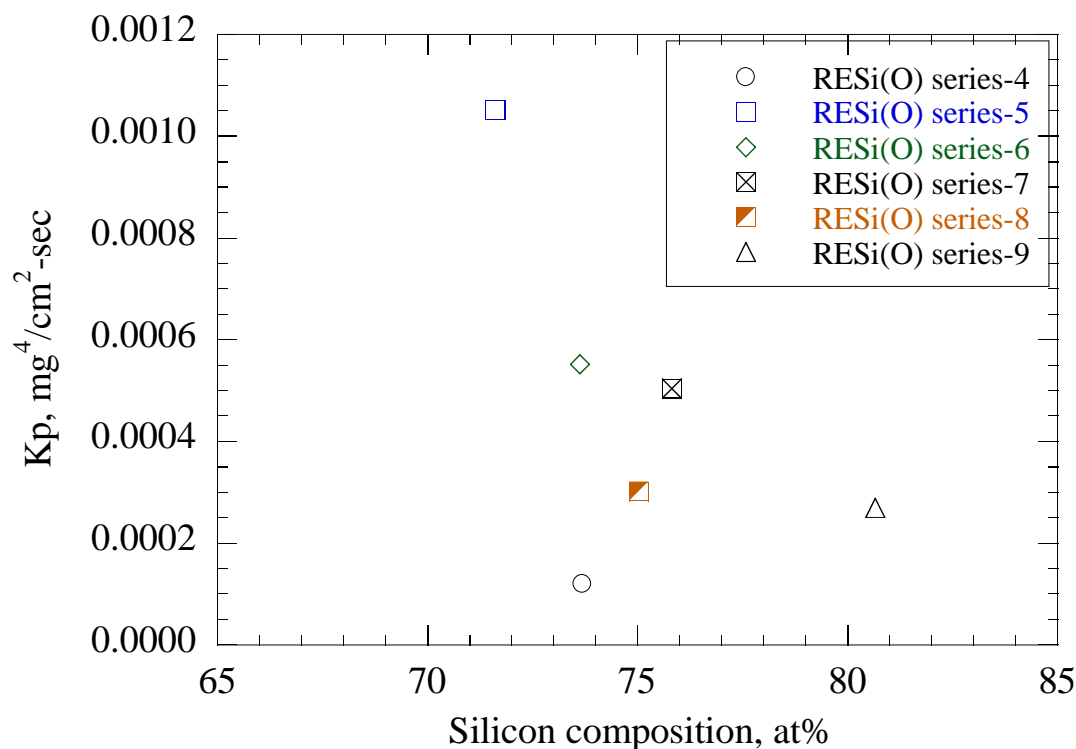


High heat flux cyclic rig tested Zr/Hf-RE-Si series  
EBC bond coats on the bond coated woven  
SiC/SiC CMCs at 1450°C in air and full steam  
environments

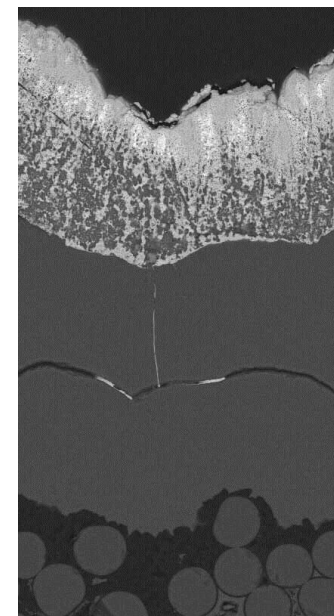


# Advanced Bond Coats for Turbine Airfoil and Combustor EBCs Developed - Continued

- 1500°C (2700°F) capable RESiO+X(Ta, Al, Hf, Zr ...) EBC bond coat compositions and related composite coatings developed for combustor and turbine airfoil applications
- Oxidation kinetics being studies using TGA in flowing O<sub>2</sub>
- Parabolic or pseudo-parabolic oxidation behavior observed

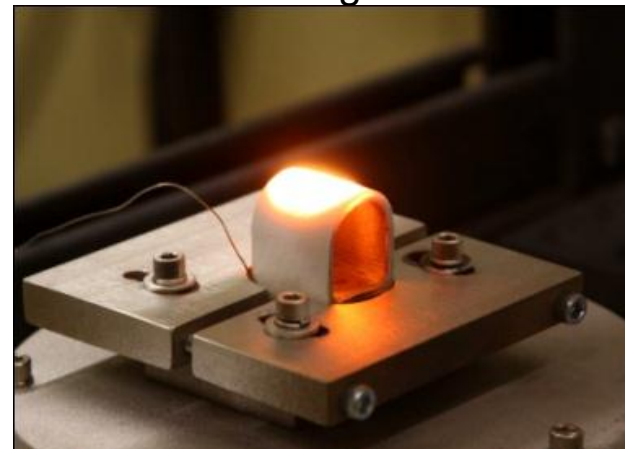
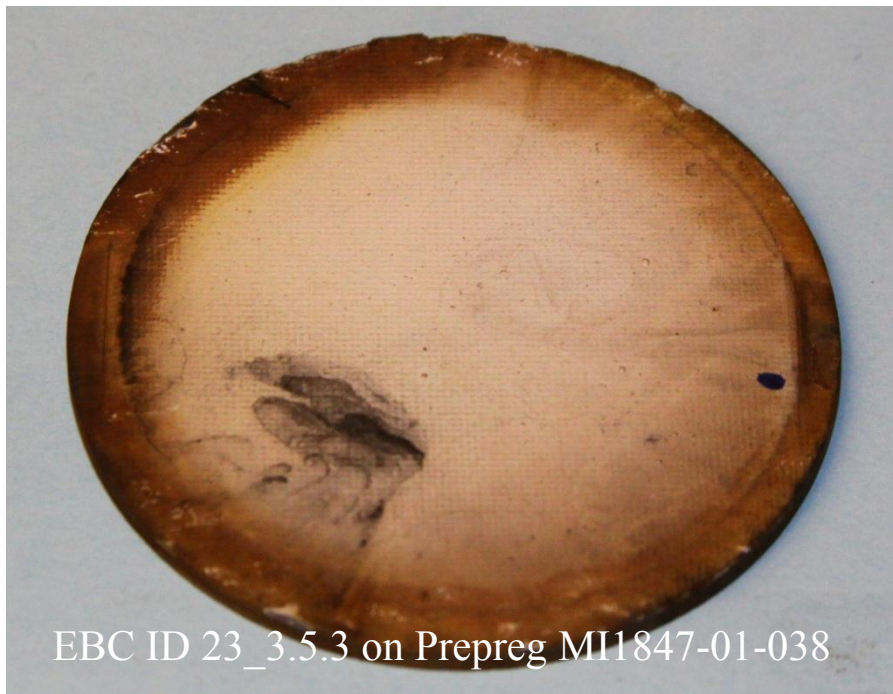


An oxidized bond coat after 1500°C 100 h creep testing



## NASA Turbine Environmental Barrier Coating Testing Developments

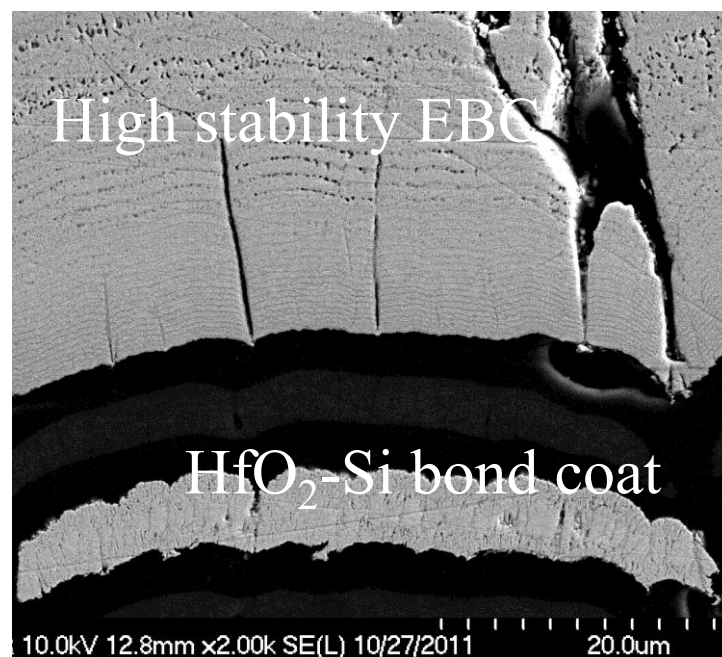
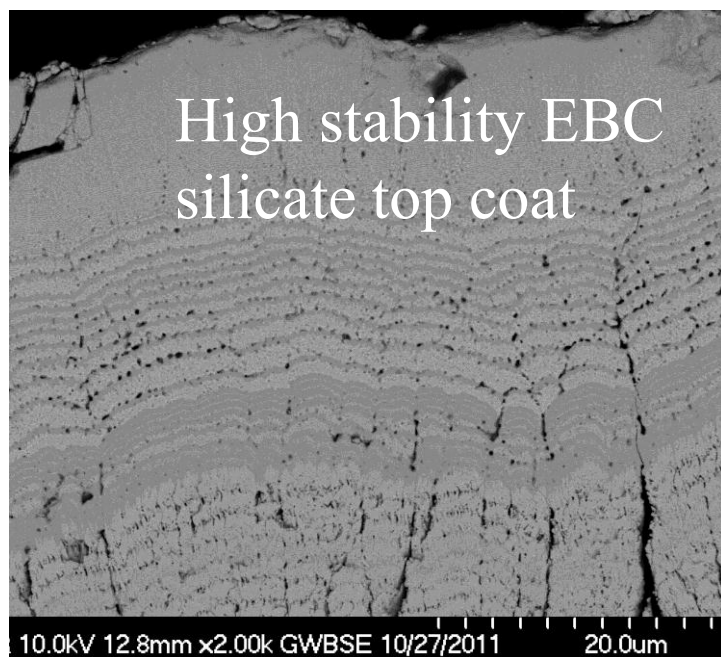
- Advanced EBC top coats tested in coupons under laser heat flux cyclic rigs up 1700°C
- Coated subelements coating tested up 1500°C under laser thermal gradient for 200 hr
- EBC systems show high stability in High Pressure Burner Rig Tests
- Low thermal conductivity of 1.2 W/m-K for optimized turbine airfoil coatings



High pressure burner rig, 16 atm, 31 hr

## NASA Turbine Environmental Barrier Coating Testing Developments

- High stability systems (Yb,Gd,Y+Hf) silicates, processed and down selected
- Processing optimization also emphasized

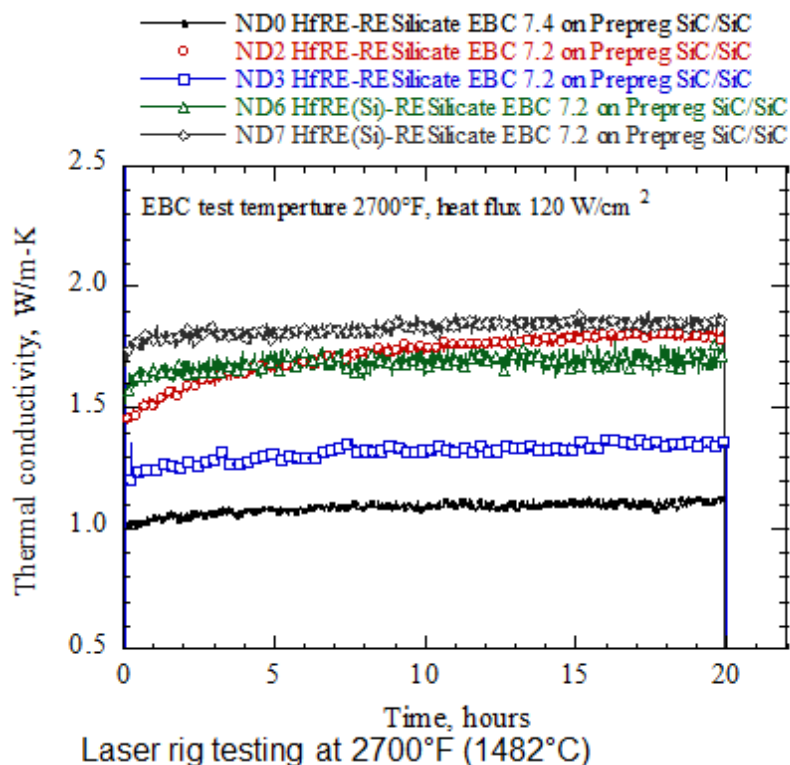


Turbine EBCs: High pressure burner rig tested at 10 atm, 2650°F

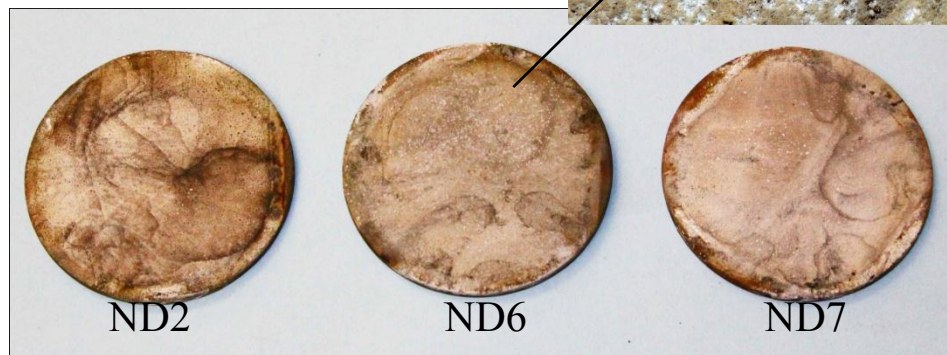


## Advanced EBC developments – Some Hybrid APS-PVD Systems and Qualification Tests

- EB-PVD  $\text{HfO}_2\text{-RE}_2\text{O}_3$  (Silicate) top coat EBC with plasma-sprayed multi-component advanced silicate sublayer EBC/ $\text{HfO}_2\text{-Si}$  bond coat systems
- Low thermal conductivity ranging 1.0 - 1.7 W/m-K
- Demonstrated high pressure environmental stability at 2600-2650°F, 12-20 atm in the high pressure burner rig



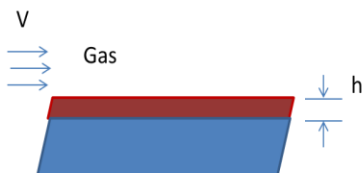
2" diameter ND3 EBC/SiC/SiC specimen after testing in the high pressure burner rig At 2600°F



High pressure burner rig tested new ND series Hybrid EBC systems coated on 2" diameter Gen II Prepreg SiC/SiC CMCs

# Understanding High Velocity Gas Flow Interactions – Columnar Structure and Toughness Considerations

- High velocity, high pressure gas impingements and shear force induced erosion in turbine engine flow condition can be of concern for low toughness coating systems
- High toughness, optimum coating density and architectures are required for durability



Shear Stress

$$\tau = \frac{F}{A} = C_D \left( \frac{1}{2} \rho V^2 \right)$$

F Shear Force

A area

$C_D$  drag coefficient

$\rho$  density

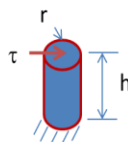
V velocity

For Ideal Gas:

$$\rho = \frac{P}{RT}$$

$$\tau = C_D \left( \frac{1}{2} \frac{P}{RT} V^2 \right)$$

If the coating is formed from columnar structures:



Bending stress at the base

$$\sigma = \frac{M c}{I} = \frac{\tau \pi r^2 h r}{\left( \frac{\pi r^4}{4} \right)} = \frac{4 \tau h}{r}$$

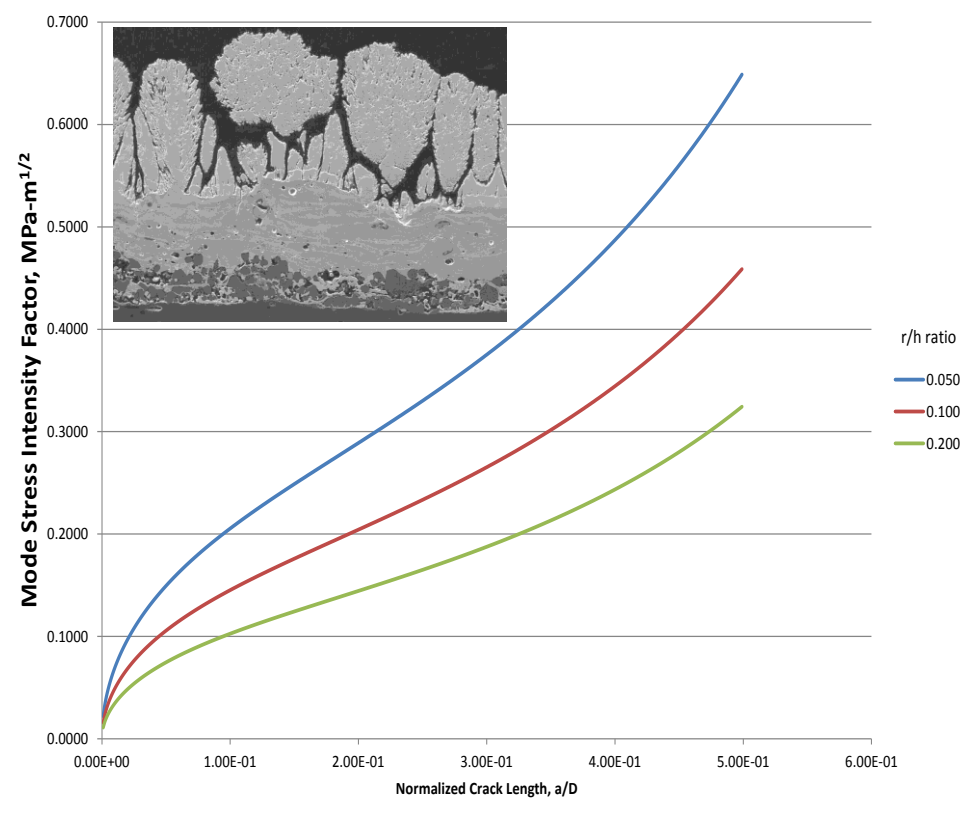
$$\sigma = \frac{4 h}{r} \left( C_D \frac{P V^2}{2 R T} \right) = \frac{2 h}{r} \left( C_D \frac{P V^2}{R T} \right)$$

For a columnar structure with defect of size a

$$K_I = \sigma \sqrt{\pi a} f(a, r)$$

$$K_I = \frac{2 h}{r} \left( C_D \frac{P V^2}{R T} \right) \sqrt{\pi a} f(a, r)$$

$K_I$  vs. Normalized crack depth

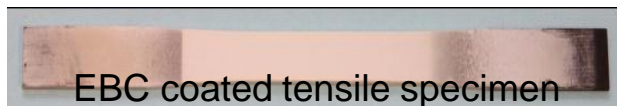


## Modeled parameters

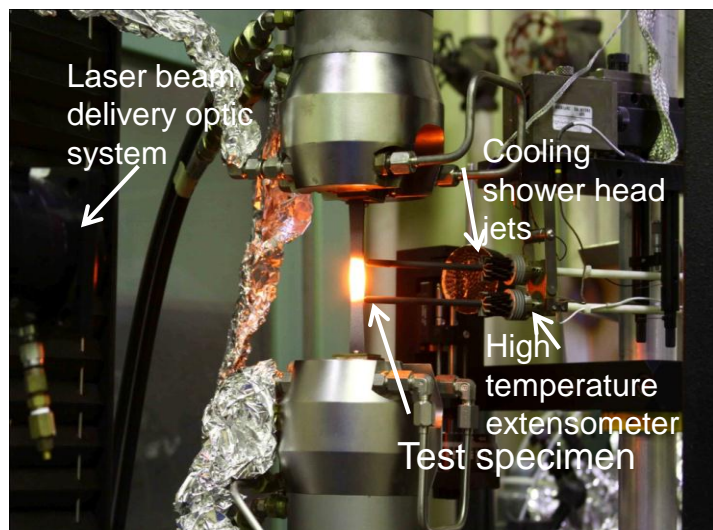
Drag Coefficient	Cd		0.4
Pressure	P, psi	750	5171068 Pa
Velocity	V		1200 m/sec
Temperature	T, F	3000	1921.039 K
Gas Constant	R		461.5 J/Kg/K
Column Height	h		0.0002 m
Column Radius	r		0.00001 m
Stress			1.34E+08 Pa

## Thermal Gradient Tensile Creep Rupture Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs

- Advanced high stability multi-component hafnia-rare earth silicate based turbine environmental barrier coatings being successfully tested for 1000 hr creep rupture
- EBC-CMC creep, fatigue and environmental interaction is being emphasized



2400°F



2400°F

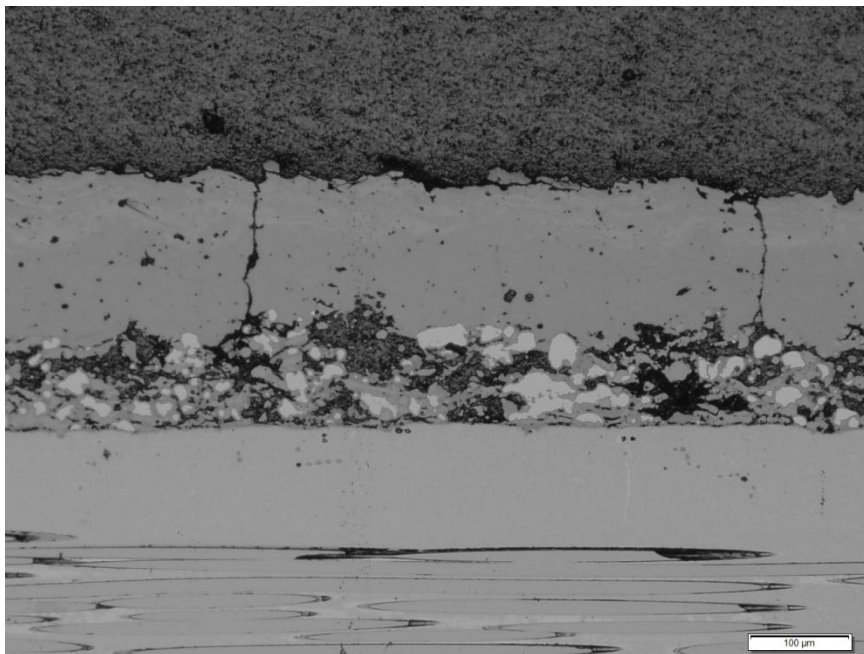
2400°F

2250°F

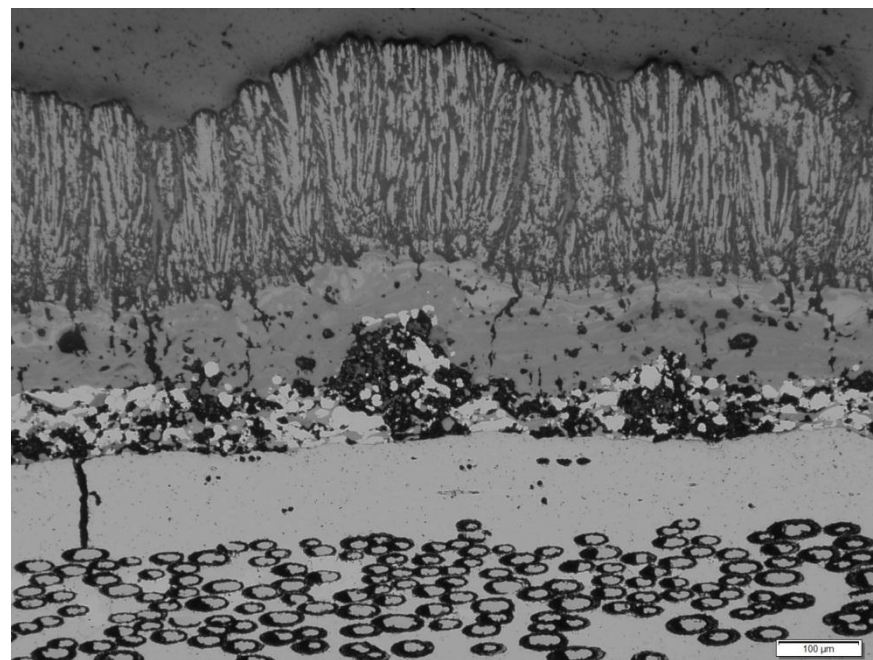


# Thermal Gradient Fatigue-Creep Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs - Continued

- Advanced environmental barrier coatings – Prepreg CMC systems demonstrated long-term EBC-CMC system creep rupture capability at stress level up to 20 ksi at  $T_{\text{EBC}} 2700^{\circ}\text{F}$ ,  $T_{\text{CMC interface}} \sim 2500^{\circ}\text{F}$
- The  $\text{HfO}_2$ -Si bond coat showed excellent durability



EBCs on Gen II CMC after 1000 hr fatigue testing

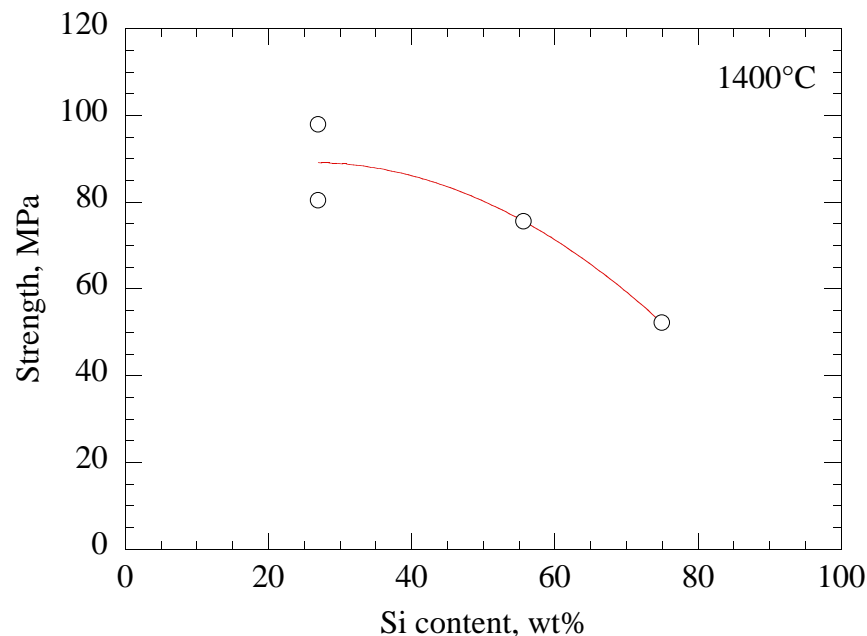
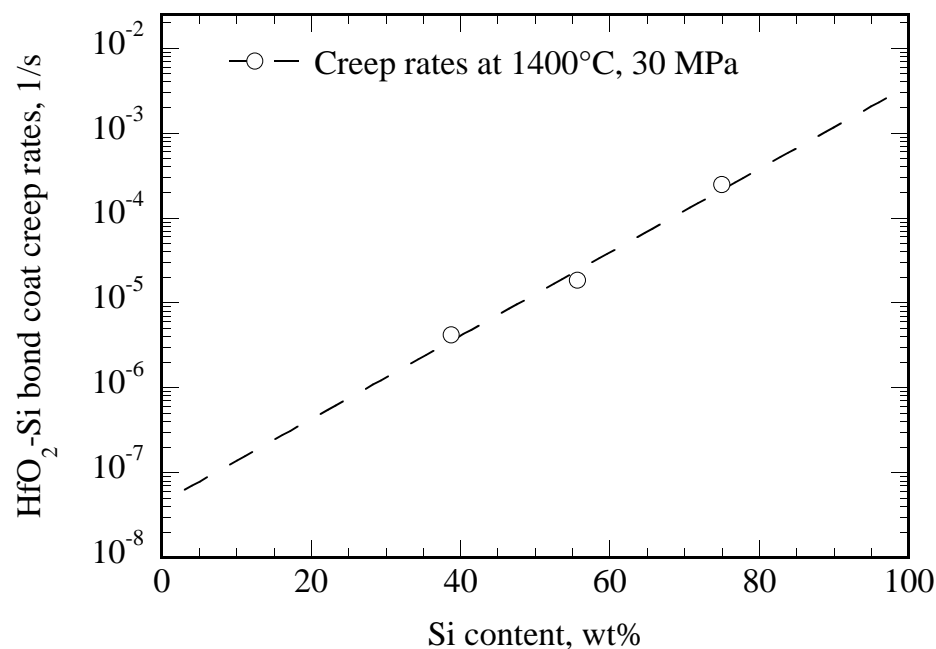


Hybrid EBCs on Gen II CMC after 100 hr low cycle creep fatigue testing



# Advanced $\text{HfO}_2$ -Si Bond Coats: Effects of Compositions on Strength and Creep Rates

- The  $\text{HfO}_2$ -Si composite coatings have high strength, and improved creep resistance at high temperatures
- Increased  $\text{HfO}_2$ - $\text{HfSiO}_4$  contents improve high temperature strength and creep resistance



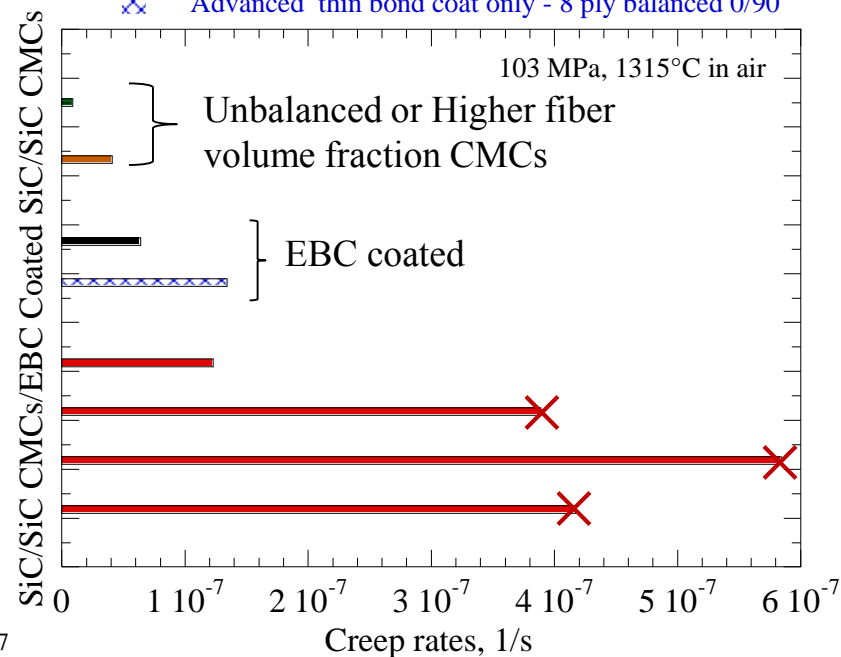
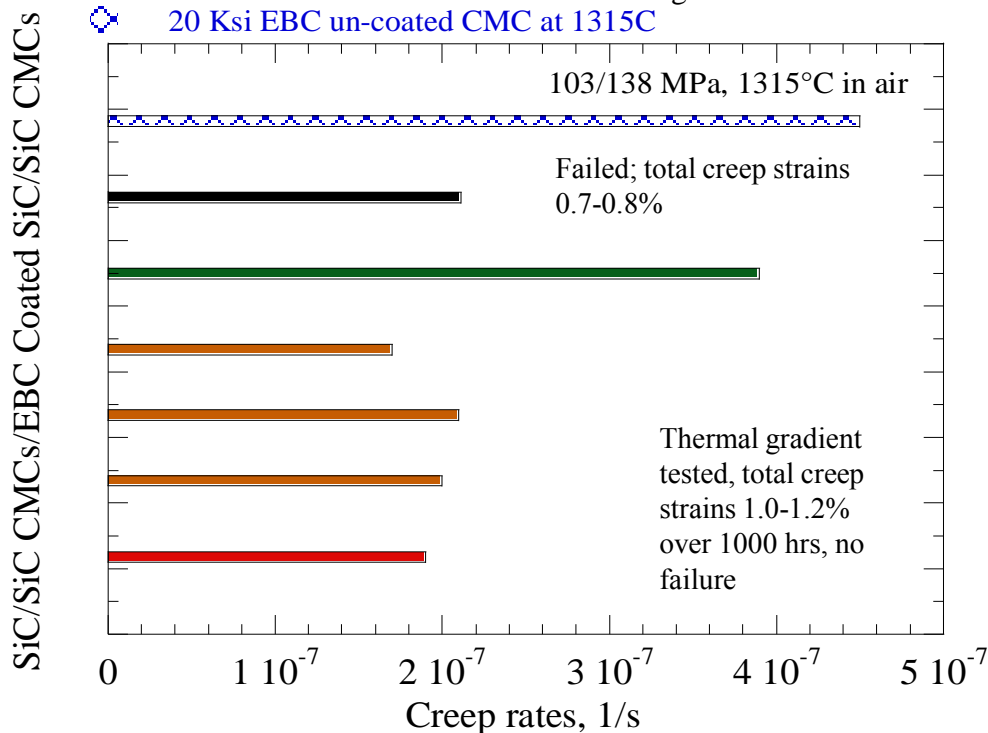
# Thermal Gradient Fatigue-Creep Testing of Advanced Turbine Environmental Barrier Coating SiC/SiC CMCs - Continued

- Effects of temperature, load, heat flux and environments (steam and combustion air) for coated SiC/SiC CMC are being investigated
- EBC coated CMCs showed improved durability

- 15 Ksi, thermal gradient\_EBC coated CMC (TCMC average 1315C)
- 20 Ksi, thermal gradient\_EBC coated CMC (TCMC average 1315C)

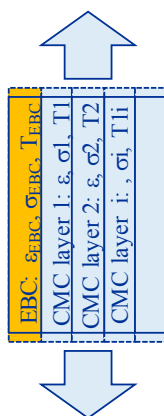
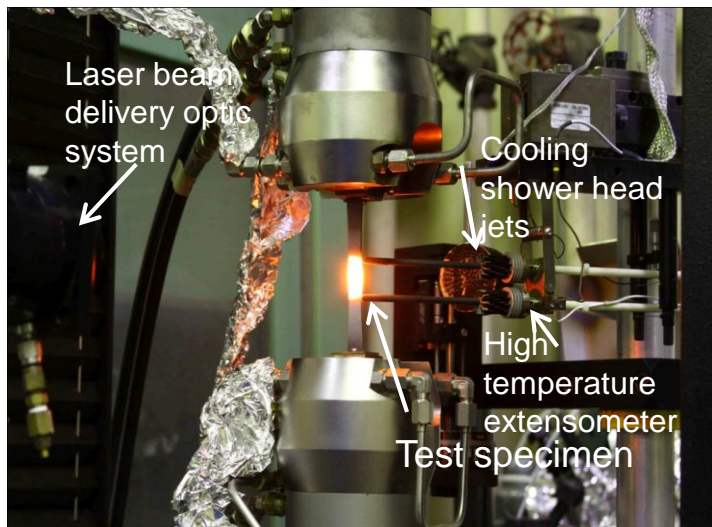
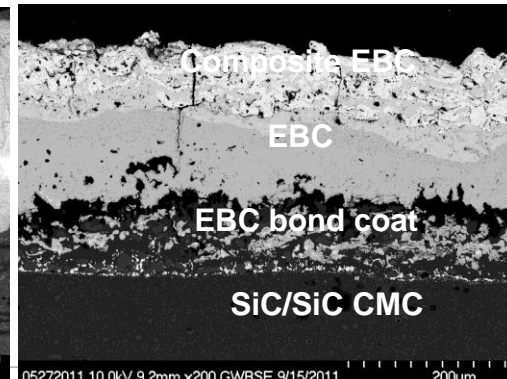
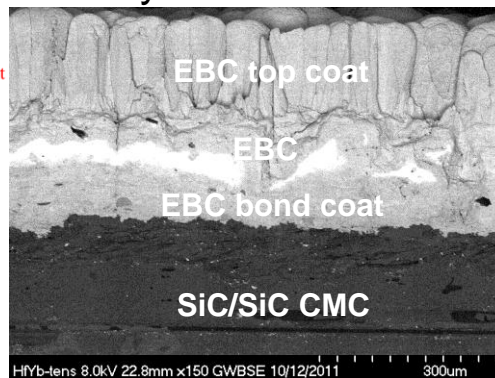
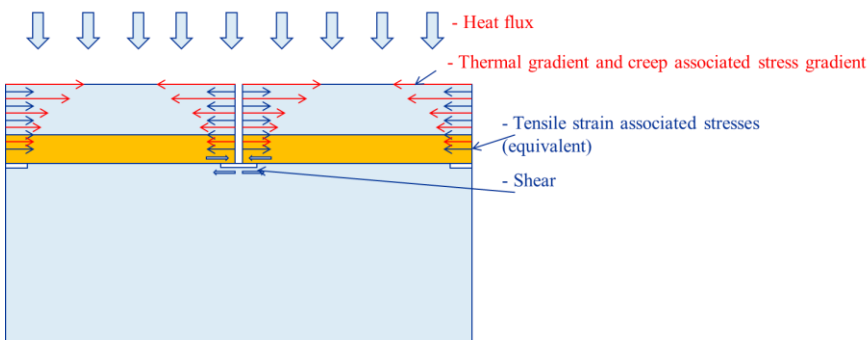
- 20 Ksi EBC coated CMC at 1315C
- 20 Ksi EBC coated CMC at 1315C Fatigue maximum stress 20 Ksi
- 20 Ksi EBC un-coated CMC at 1315C

- Gen II MI SiC/SiC 8 Ply balanced 0/90
- Gen II MI SiC/SiC 6 ply unbalanced 0/90
- CVI Woven SiC/SiC
- Advance EBC system coated 8 ply balanced 0/90
- Advanced thin bond coat only - 8 ply balanced 0/90

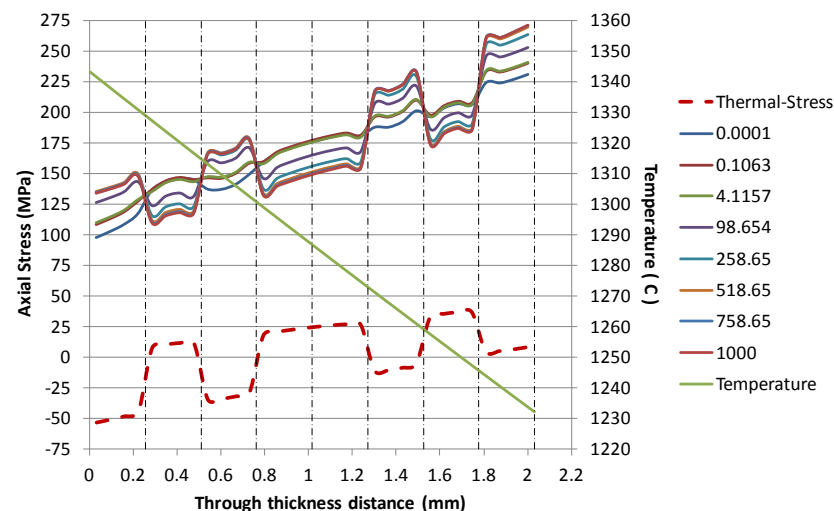


# EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling

- An equivalent stress model is established for EBC multicrack stress intensity modeling: emphasize creep, thermal gradient and stress rupture interactions
- Benchmark failure modes established in EBC systems



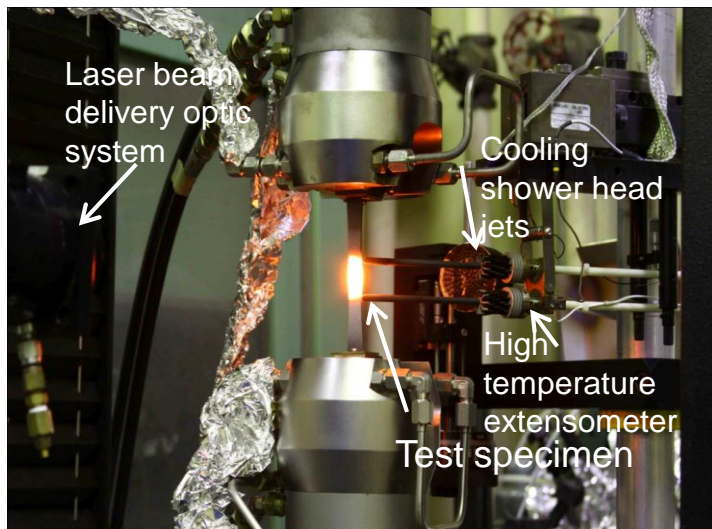
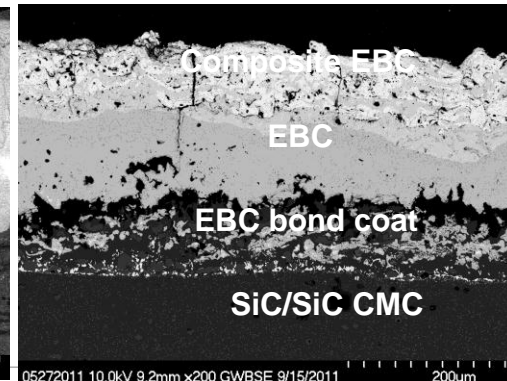
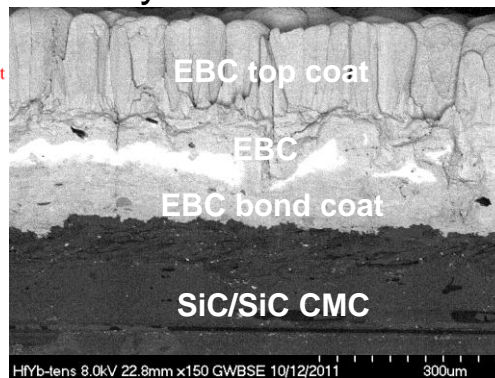
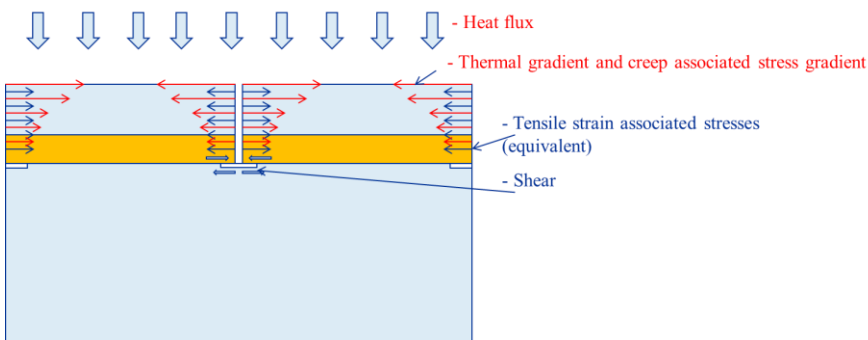
Through the thickness axial stress profile at different time



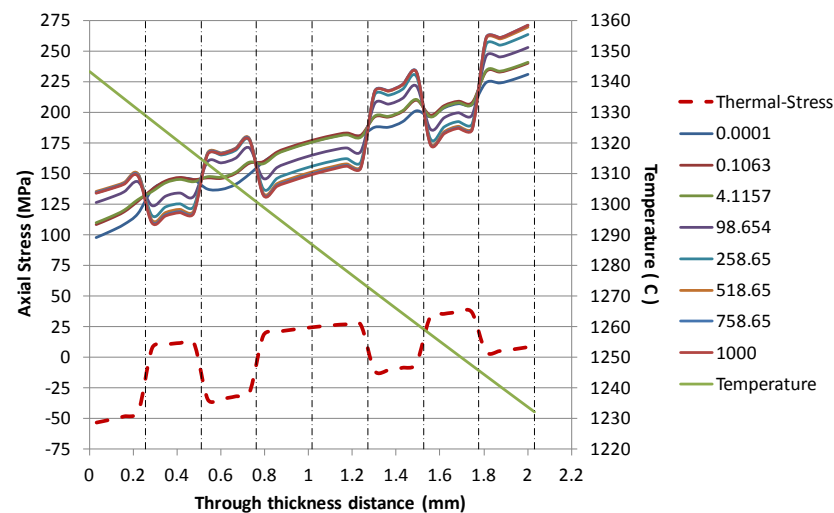
Stress gradients in Prepreg SiC/SiC CMC substrates under thermal gradient + mechanical creep loading

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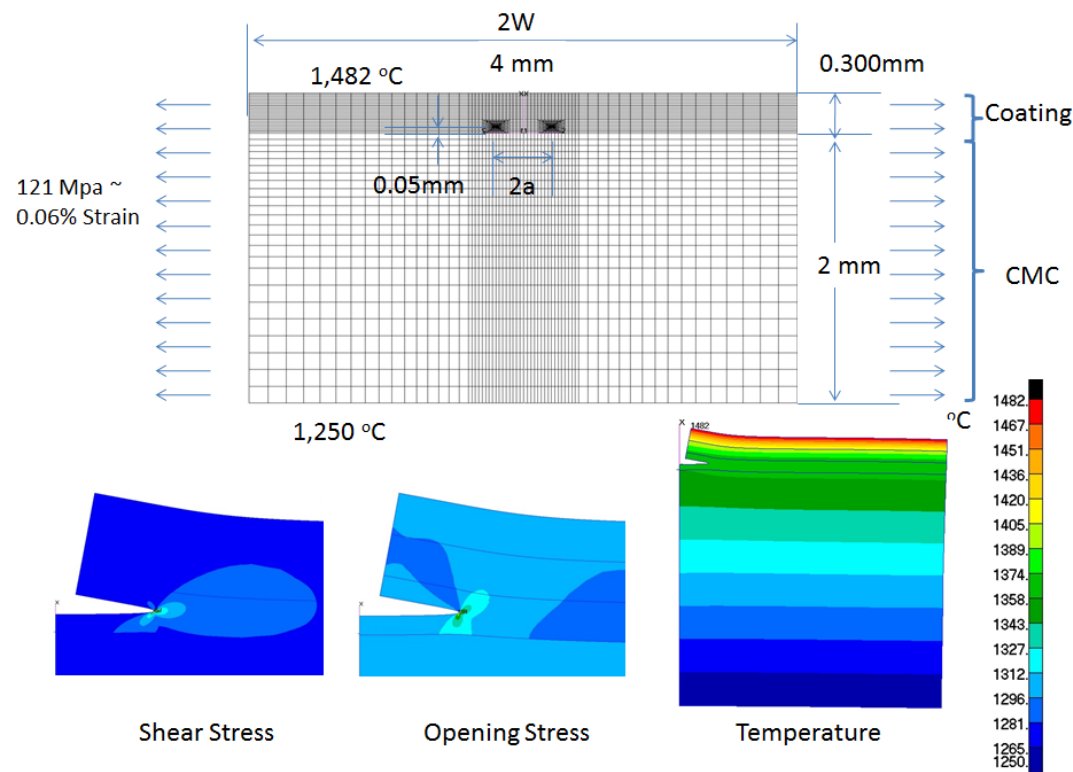
Through the thickness axial stress profile at different time



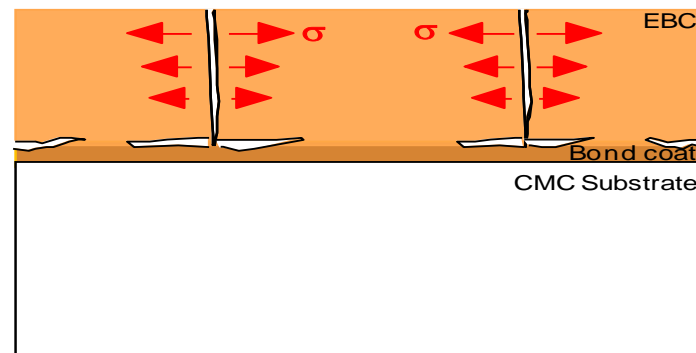
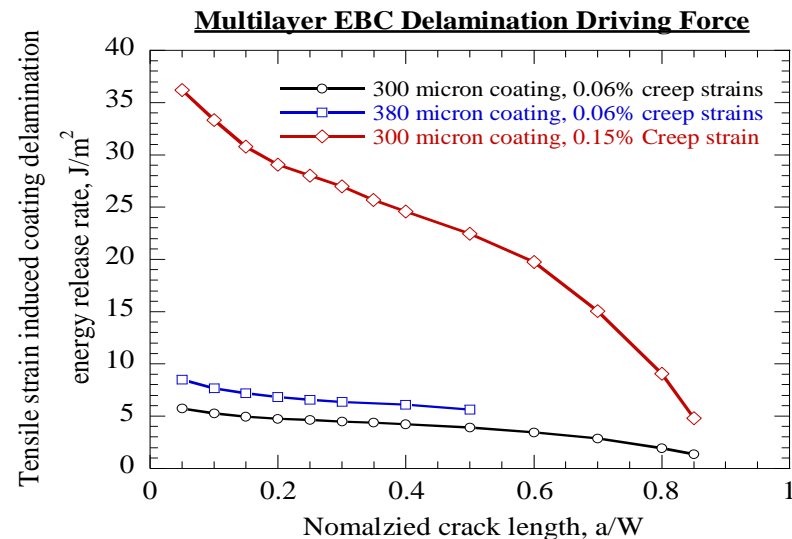
Stress gradients in Prepreg SiC/SiC CMC substrates under thermal gradient + mechanical creep loading

# EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling - Continued

- EBC surface and delamination cracking under heat flux, thermal gradient tensile creep loading ( $T_{\text{surface}} = 1482^{\circ}\text{C}$ ;  $T_{\text{back}} = 1250^{\circ}\text{C}$ )
- Delamination failure driving forces determined
- Uniform displacement case: effect of coating thickness



Finite Element Analysis (FEA) Modeling

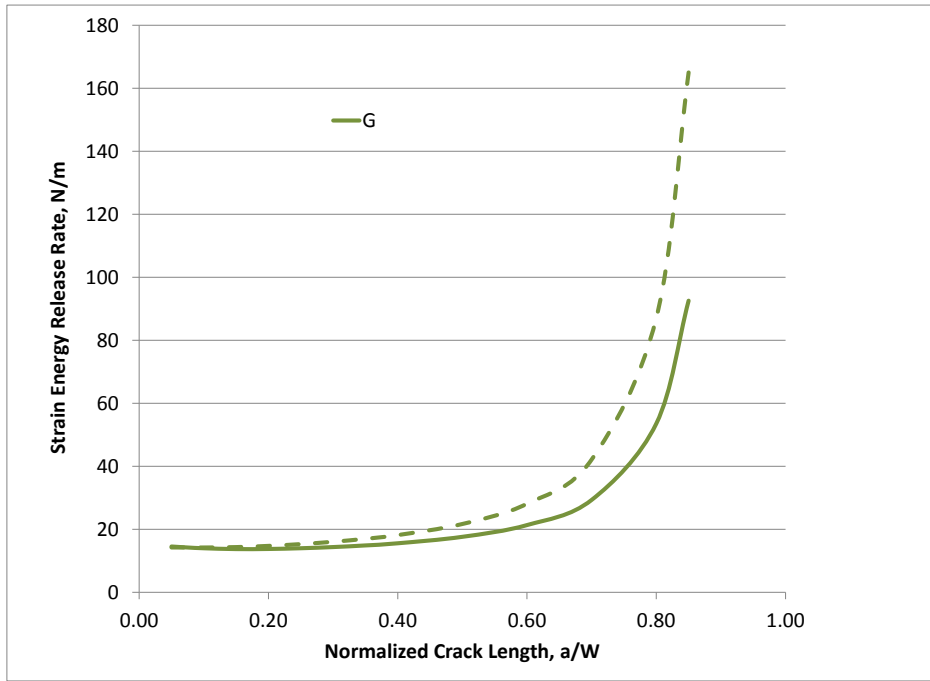
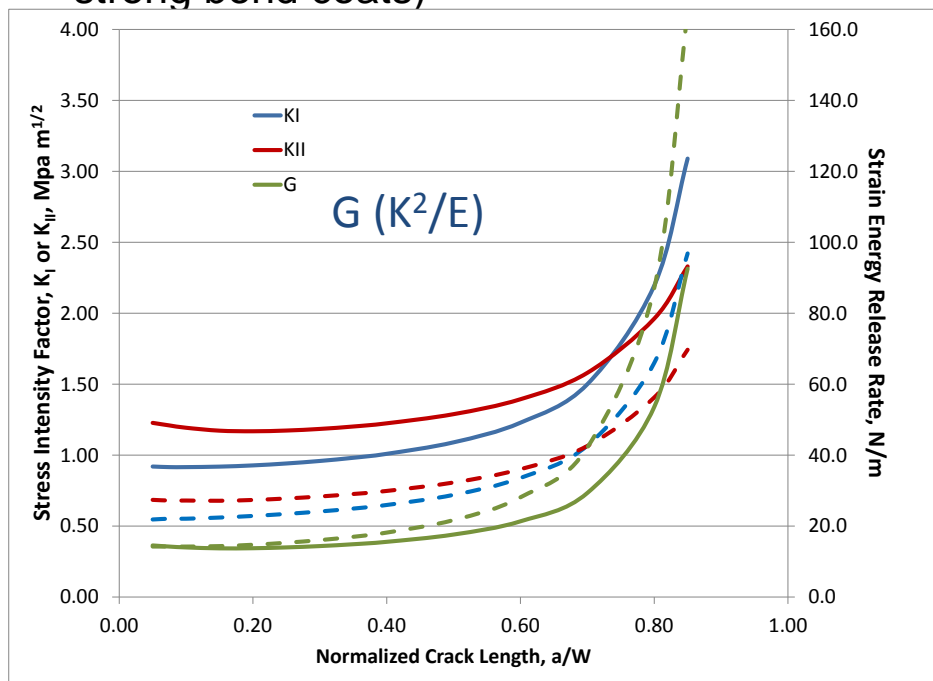


EBC degradation under tensile creep loading

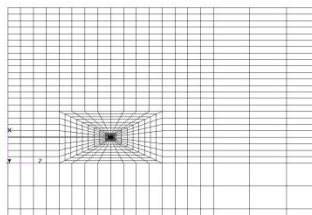


# EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling – Bond Coat Stiffness Effect

- Delamination driving forces: uniform remote applied stress case, 0.300 mm thickness coating with  $\sim 0.06\%$  total strain
- Effect of bond coat elastic modulus:  $E=150$  GPa vs.  $E=50$  GPa
- Strong bond coats expected to have less creep damage (lower strain energy release rate  $G$  for strong bond coats)



Solid Lines-strong bond coat  
 $E=150$  GPa EBC  
 $E=150$  GPa bond coat

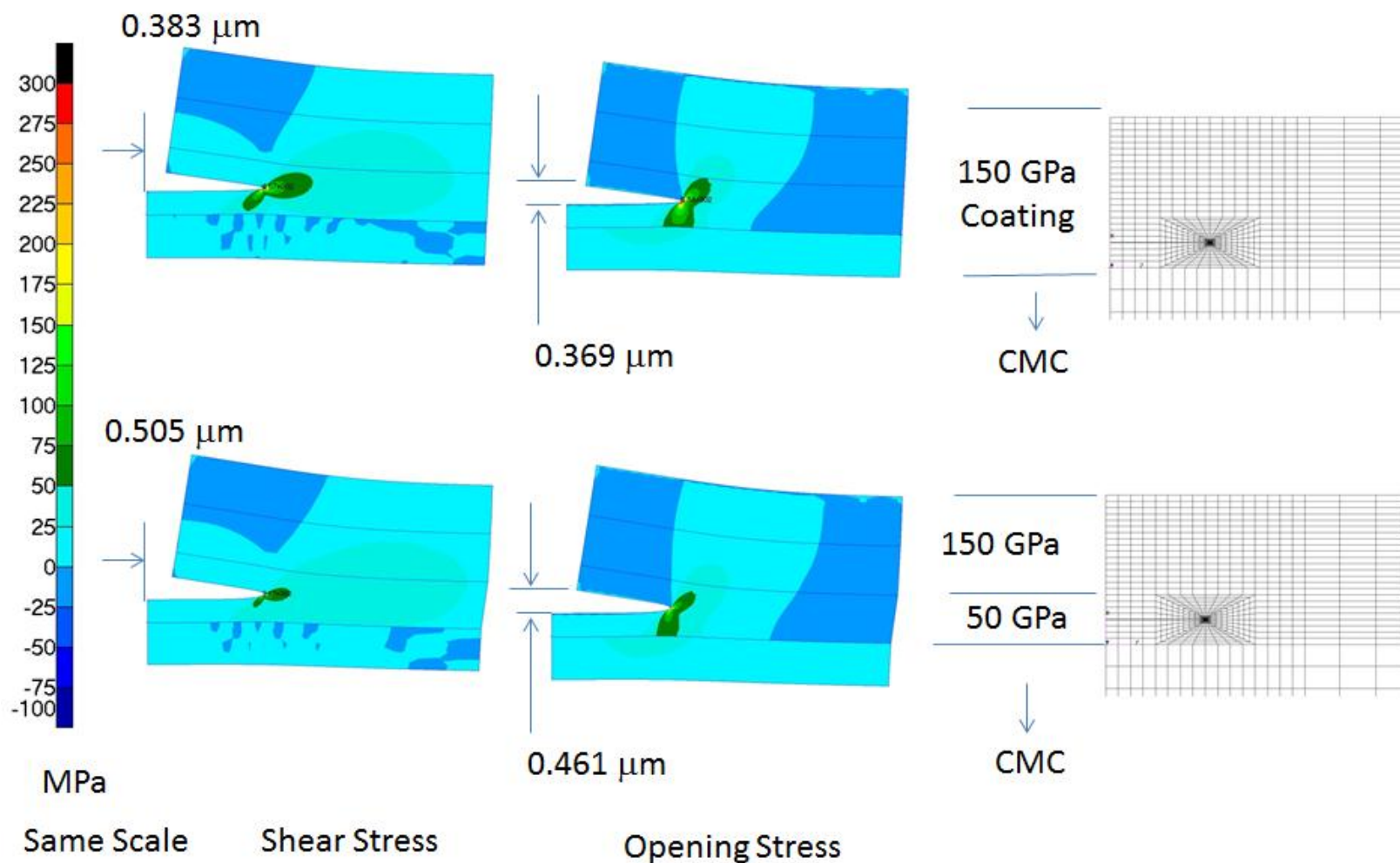


$E=150$  GPa  
 $E=50$  GPa

Dashed Lines: Soft bond coat

## EBC-CMC Thermal Gradient Creep Rupture and Delamination Modeling – Bond Coat Stiffness Effect

- Advanced EBCs designed with higher high temperature strength and stiffness to improve creep, fatigue, and cyclic durability





## The Advanced EBC on SiC/SiC CMC Turbine Airfoils Successfully Tested for Rig Durability in NASA High Pressure Burner Rig

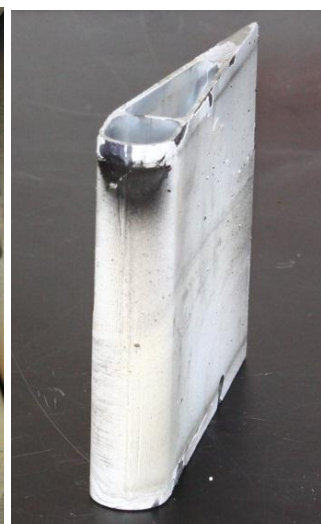
- NASA advanced EBC coated turbine vane subcomponents tested in rig simulated engine environments (up to 240 m/s gas velocity, 10 atm), reaching TRL of 5
- Turbine EBCs generally intact (some minor partial coating top coat spalling for the Prepreg MI SiC/SiC vane)
- Some minor CMC vane degradations after the testing



EBC Coated CVI SiC/SiC vane after 31 hour testing at 2500°F+ coating temperature



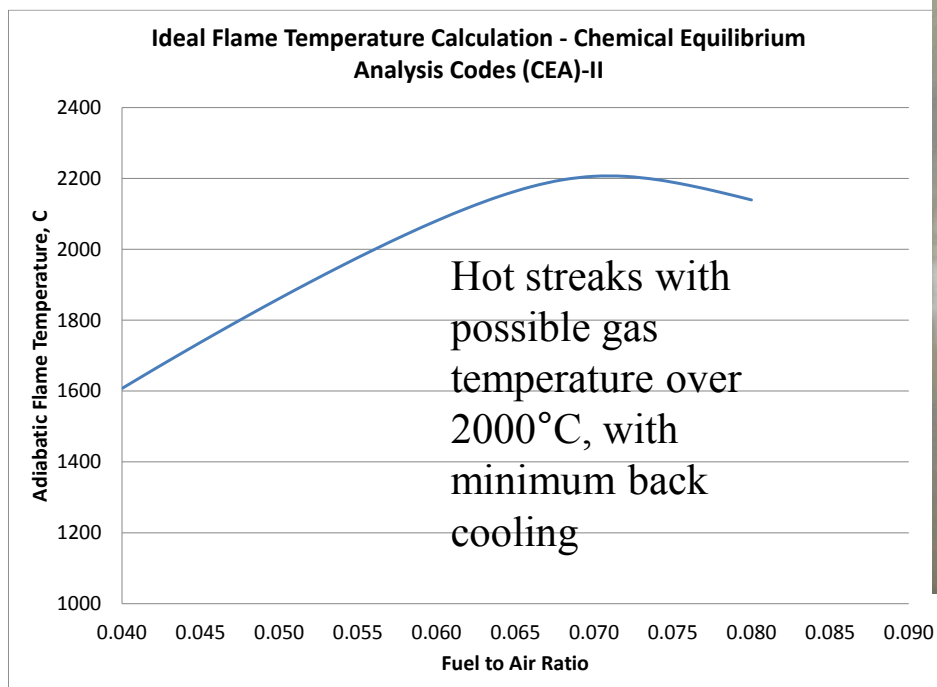
EBC Coated Prepreg SiC/SiC vane after 21 hour testing at 2500°F



EBC Coated Prepreg SiC/SiC vane tested 75 hour testing at 2650°F

## The EBC Coated SiC/SiC CMC Combustor Liner Successfully Demonstrated for Rig Durability in NASA High Pressure Burner Rig (First Inner Liner Processed at Sulzer with Triplex Pro)

- Tested pressures at 500 psi external for outliner, and up to 220 psi inner liners in the combustion chamber (16 atm), accumulated 250 hours in the high pressure burner rig
- Average gas temperatures at 3000°F (1650°C) based on CEA calculations, the liner EBCs tested at 2500°F (1371°C) with heat fluxes 20-35 W/cm<sup>2</sup>, and the CMC liner component at 1800-2100°F (~1000-1100°C)



Some minor coating spalling at hot streak impingement

## Summary

- **Durable EBCs are critical to emerging SiC/SiC CMC component technologies**
  - The EBC development built on a solid foundation from past experience
  - Advanced EBC processing and testing capabilities significantly improved, helping more advanced coatings to be realized for complex turbine components
  - A new series of EBC and bond coat compositions developed for meeting SiC/SiC CMC component performance requirements and long-term durability, establishing expanded scientific research areas
  - Better understood the coating failure mechanisms, and helping developing coating property databases and life models, aiming at developing higher stability, higher strength EBC and bond coats
  - Emphasized thin coating turbine and combustor EBC coating configurations, demonstrated component EBC technologies in simulated engine environments
  - *Continue the coating composition and architecture optimization and developments to achieve 1482-1650°C capability, targeting uncooled and highly loaded components*
    - *The component and subelement testing and modeling*
  - *Understand EBC-CMC degradation and life prediction under complex thermal cycling, stress rupture/creep, fatigue, and environmental integrations*

## **Future Directions and Opportunities**

- **High stability turbine airfoil and combustor coating system development continues to be a high priority**
  - Emphasize advanced composition development, optimization, new processing and modeling capabilities
  - Reduce recession rates, improve the temperature stability and environment resistance, such as in CMAS environments
  - Significantly improve the interface stability and reduce reactivity
  - Low thermal conductivity
- **Advanced environmental barrier coatings with significantly improved thermal and mechanical load capability is required**
  - Significantly improve the coating strength and toughness
  - Better understand and improve creep, fatigue, and environment interactions
  - Design and demonstrate long-term high heat flux cyclic stability
- **Materials and component system integration**
  - Optimize and test coatings with components and SiC/SiC substrates
  - Enhance functionality with embedded sensing and self-healing capability
  - Integrate with virtue sensors and real time life predictions
- **Laboratory simulated high heat flux stress, environment testing and life prediction methodology development, validating model developments**